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ERDEC-TR-440

DENSITY MEASUREMENTS OF SEVERAL GRASS POLLENS

Anna Wong

RESEARCH AND TECHNOLOGY DIRECTORATE

September 1997

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RESEARCH, DEVELOPMENT & ENGINEERING CENTER
U.S. ARMY CHEMICAL AND BIOLOGICAL DEFENSE COMMAND

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DEPARTMENT OF THE ARMY
U.S. Army Edgewood Research, Development and Engineering Center
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Procedures for making density measurements on an autopycnometer are described. Data from five samples of grass pollen (Bahia, Bermuda, Kentucky/June Blue, Corn and Meadow Fescue) are included in the report.

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PREFACE

The work described in this report was authorized under Project No. 10262384A553, Nonmedical CB Defense. This work was started in June 1996 and completed in September 1996.

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1. BACKGROUND

Materials such as pollens, molds, fungi, smuts and bacteria fluoresce when exposed to ultraviolet radiation. A database of these fluorescent characteristics is useful in comparing naturally occurring aerosols with artificial aerosols. Fluorimeters can accomplish this task but the samples are usually suspended in some type of buffer solution. There are inherent problems connected with a liquid suspension.

- The act of suspension may require the particles be treated to make them more hydrophilic (as with pollens);
- The liquid may actually change the nature of the particles being studied (as with molds and bacteria);
- The spectral data may reflect the fluorescence of the buffer solution; and
- The sample may settle as the measurements are being performed causing the concentration to vary.

To avoid these problems, a stead flow of dry particles through the fluorimeter, or other measurement device, can replace the solution suspension. As an added bonus, this allows better control of the temperature and humidity of the sample. Particles leaving the system will travel to an aerosol particle sizer (APS) that measures the particle diameter and size distribution. The concentration is then calculated from this data.

The APS uses a technique known as aerodynamic time of flight developed by Dr. Barton E. Dahneke[1]. The device accelerates the particles to sonic speeds and measures the time it takes to travel between two laser beams when that force is no longer applied. The device calculates the particle size from this measurement and the density. Therefore the accuracy of the density measurement is crucial to this calculation.

[1] "Aerosizer Technical Manual," Amherst Process Instruments, pp TM2, Hadley, MA, undated.

2. INTRODUCTION

Theoretically, the density of a material is simply the mass per unit volume. Because most particles are rarely homogeneous or anhydrous, the practical measurement of the density is not a simple matter. Irregular shaped particles, as shown in Figure 1[2], occupy a greater volume, even if compressed, than their actual volume. And, most powders will absorb or adsorb water causing significant variations in the mass and volume of a sample, as shown in Figure 2[3]. The raw data reflects Kaolinite tested as shipped from the manufacturer. The normalized data uses the recalculated mass from the last point of the raw data. And the pre-dried data was taken on a sample that was dried for 12 hours at 250°C. To obtain an accurate measure of the volume a device called a pycnometer is used. To control the humidity of the sample, careful handling and preparation procedures are observed.

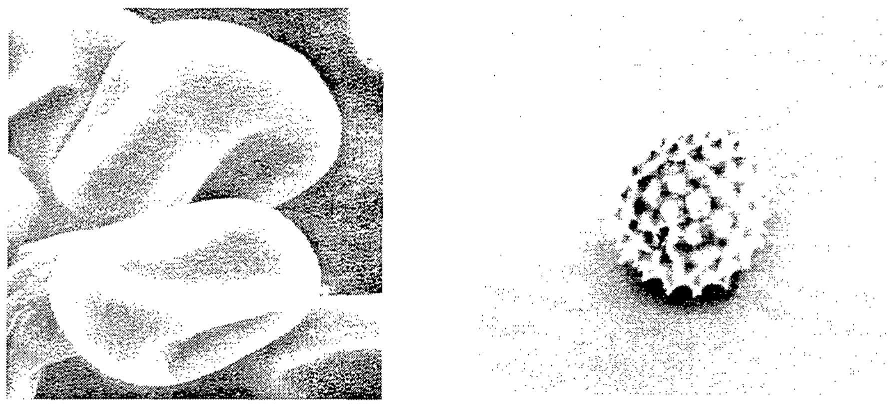

Figure 1. Bermuda grass pollen and ragweed pollen at 1000x magnification

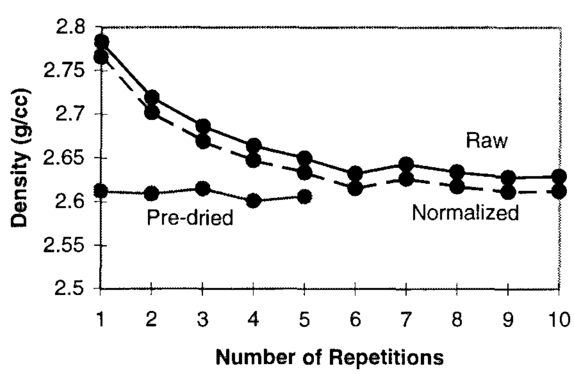

Figure 2. Density of Kaolinite.

[2] McCrome and Delly, "The Particle Atlas; and Encyclopedia of Techniques for Small Particle Identification," Ann Arbor Science Publishers, Ann Arbor, MI, 1980.

[3] "Instruction Manual for Autopycnometer 1320," Micromeritics, pp 2-2, 3-3, 4-6, Norcross, GA, 1 March 1984.

3. THE INSTRUMENT

A pycnometer is a device that uses helium to measure the volume that a sample displaces. Since helium is a small atom it can fill and seep into small irregular features. The instrument used in this report was a helium pycnometer manufactured by Micromeretics Instrument Corporation (model 1320). The precision of each measurement is ± 0.001 cc. The accuracy from measurement to measurement is given as ± 0.02 cc by the manufacturer[3]. The accuracy depends a great deal on the care taken in preparing the sample which will be discussed in a later section.

3.1 Initial Check Out Procedures

3.1.1 Purging

Purging the instrument is necessary when the instrument has not been operated for a long duration. It evacuates the air from and establishes helium in Chambers A, B, C, D, and E (see Figure 3) within the pycnometer.

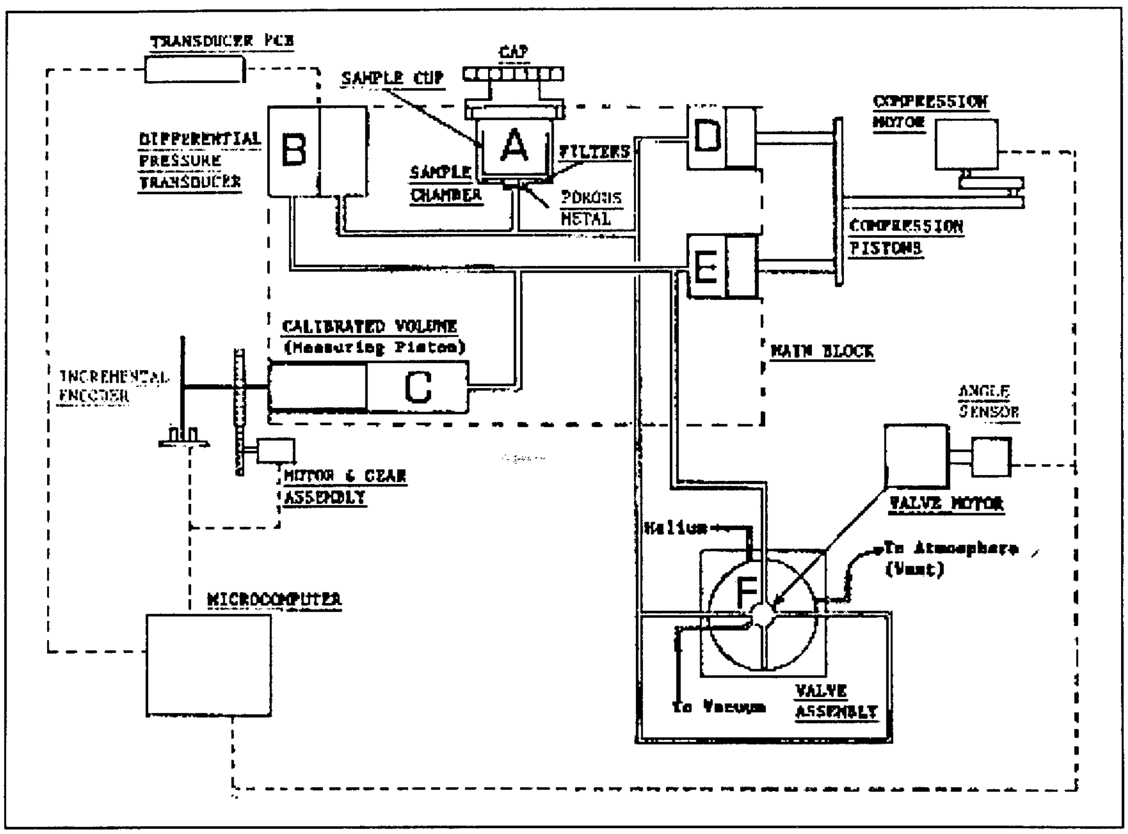

Figure 3. Vacuum/Helium flow diagram[3] for Model 1320 Autopycnometer.

The instructions for purging are given below.

1. Give the large knob/lid a firm, quarter turn in the counterclockwise direction. This will release the lid and expose the sample cavity.

2. Check the following items:
    - O-rings should be lubricated with vacuum grease and the grease should be clean and free of debris.
    - A fiber filter should cover the bottom of the sample cavity. The fiber should be clean and flat.
    - A thin wire grid (under the filter) and the small metal frit (beneath the grid) should be seated flat underneath the filter.
3. Replace the lid by turning the it a firm, quarter turn in the clockwise direction. This reestablishes the vacuum seal in the sample cavity.
4. Open the valve for the helium. Check that the regulator is set to 5 psig. (NOTE: More than 10 psig will damage the instrument.)
5. Turn on the vacuum pump. Check the oil level. Level should reside between the minimum and maximum levels.
6. Turn on the power to the autopycnometer.
7. Set the WEIGHT thumbwheel to 00.000 grams.
8. Set the EVAC thumbwheel to 1 minute.
9. Depress the RUN switch.
10. Wait for the LOAD light to illuminate.
11. Repeat steps 9 and 10 twice.

THE INSTRUMENT IS NOW SUFFICIENTLY PURGED!!

3.1.2 Nullifying

Setting the null on the pycnometer negates the volume of the container. Once set for a particular sample container, that information will remain in memory until the process is performed again.

Matched sets of sample containers may be purchased from the manufacturer which have the same volume as well as equal weight of the lid to the matched lid and the cup to the matched cup. Matched containers were used to gather the data in this report since it provides the convenience of preparing one sample while running a second. Note that only one of the matched containers was used to null the pycnometer.

The instructions for nulling the sample container are given below.

1. Give the large knob/lid a firm, quarter turn in the counterclockwise direction. This will release the lid and expose the sample cavity.
2. Put the sample container and lid into the sample cavity and reseal the cavity with the lid.
3. Set the WEIGHT thumbscrew to 00.000 grams.
4. Set the EVAC thumbscrew to 3 minutes.

THE INSTRUMENT IS NOW NULLED.

3.1.3 Calibration with a Standard

Calibration is performed to check for mechanical difficulties such as insufficient evacuation, dirt on the filter, broken o-ring, improper seal at the lid, etc. A sphere is used for this procedure, and the square of the volume of the sphere is entered into the pycnometer. The weight of the sphere is not used because it is easier to produce a sphere with a consistent surface than a homogeneous sphere.

The instructions for calibrating the pycnometer with a standard sphere is given below.

1. Give the large knob/lid a firm, quarter turn in the counterclockwise direction. This will release the lid and expose the sample cavity.
2. Place one of the available standards into the sample container.
3. Put the sample container and lid into the sample cavity and reseal the cavity with the lid.
4. Set the WEIGHT thumbscrew to the square of the volume of the standard. (See Figure 4.)
5. Set the EVAC thumbscrew to 3 minutes.
6. Depress the RUN switch.
7. Wait for the LOAD light to illuminate.

THE INSTRUMENT IS NOW CALIBRATED.

PART NUMBER	DIAMETER		VOLUME (cm^3)	VOLUME^2 ($(cm^3)^2$)
	NOMINAL (in)	ACTUAL (cm)		
1331/25607/00	9/16	1.42875±0.00003	1.527	2.332
1331/25608/00	11/16	1.74625±0.00003	2.788	7.773

Figure 4. Standard spheres for calibration[3].

4. SAMPLES

All the samples used in this report were purchased from Greer Laboratories. Laboratories of this type provide extracts to doctors for the determination and treatment of allergies. The sample here, however, are the clean, dry allergens used to create these extracts.

The allergens purchase are of two types: pollens and non-pollen. Pollens come defatted and natural state. Defatting is generally done to allow easy wetting in solution. Defatted samples are normally processed with high grade acetone, which removes the fat and dehydrates the sample. Since the principle use of these samples will be in a dry aerosol form, defatting was not necessary. A list of the pollens available for testing is shown in Figure 5.

COMMON NAME	BOTANICAL NAME	GREER LABORATORY LOT CODE
Bahia	Paspalum notatum	94GG231-7
Bermuda	Cynodon dactylon	24EE2-9B
Blue, Kentucky/June	Poa pratensis	24EE16-6B
Corn	Zea mays	75FF149-8
Fescue, Meadow	Festuca elatior	24GG14-5B
Johnson	Sorghum halepense	57HH15-7B
Rye, Italian	Lolium mutiflorum	51FF25-6
Timothy	Phleum pratense	66HH28-6

Figure 5. Samples from Greer Laboratories.

SAMPLE PREPARATION

As noted previously, sample preparation is a very important part of getting accurate density data. Some powders will absorb or adsorb water. If nothing is done to remove the water, the measurement will vary with the moisture in the sample. This is especially a problem if the sample was refrigerated, since coming to room temperature will cause it to pick up moisture from the air.

"Dry" samples that are not relieved of this water will give a false density reading. However, given enough consecutive measurements, the helium purge will slowly remove the moisture and the density will approach a steady number. This number must be adjusted by the new sample weight (without water) to approach the actual density. This approach waste time and gas.

To eliminate this problem the sample can be heated to temperatures between 100 to 200°F. The temperature and duration is dependent on the sample. For samples that cannot endure the heat, such as egg albumin, prolonged exposure to desiccant in a drying cabinet is necessary. Most of the samples will be dried in an oven and then transferred to a desiccant cabinet to cool. The temperature and duration will be noted with the data.

5. PROCEDURE

A schedule had to be laid out to provide optimal use of time while producing repeatable data. Several factors come to play in this schedule: the drying time, cooling time and the length of each trial.

First several samples were run to determine the minimum temperature and drying time required to obtain sample volumes within the ±0.02 cm^3 accuracy of the machine. After repeated trials, it was determined that drying for one hour in 200°F oven would be sufficient. At temperature lower than 150°F the drying time becomes longer. At temperatures higher than 250°F an unpalatable smell of scorched pollen drifts over the lab area.

The other determining factor for the schedule was the length of each trial. The pycnometer can be set for an evacuation of 1 to 9 minutes, if run automatically. Trials of 5 minutes were suggested by the manufacturer as a good starting point. Since the data for three trials does fall within the accuracy of the machine. All the trials were performed with a 5 minute evacuation. With this evacuation, a single trial requires approximately 24 minutes to complete before the pycnometer is ready for another trial.

The procedure for each sample is listed below.

1. Clean and dry the sample containers. >> Note the container. (A or B)
2. Load the pollen into the container. >>Note the common name of the pollen.
3. Place the container in a preheated oven. >>Note the time and temperature.
4. Remove the container from the oven. >>Note the time.
5. Cool the container and pollen in a drying cabinet with desiccant for 0.5 hours.
6. Weigh the container with the pollens. >>Note the combined weight.
7. Subtract the weight of the container from the combined weight. >>Note the weight of the sample.
8. Set the WEIGHT thumbwheel on the pycnometer to the sample weight.
9. Set the EVAC thumbwheel on the pycnometer to 5 minutes.
10. Press RUN.

11. When LOAD light is illuminated, the DENSITY will be displayed. >>Note the density of the sample.
12. Repeat step 10 and 11 twice.

With the availability of two sample containers, a sample could be in preparation while another would be in the pycnometer. Figure 6 shows the optimal schedule for just such conditions. In the data, the container used will be denoted by a dot for container B.

	CONTAINER A		CONTAINER B
0:00	BAKE	<LOAD>	
0:30			
1:00	COOL	<WEIGHT> <LOAD>	
1:30	TRIAL 1		BAKE
2:00	TRIAL 2		
2:30	TRIAL 3	<CLEAN&LOAD> <WEIGHT>	COOL
3:00	BAKE		TRIAL 1
3:30			TRIAL 2
4:00	COOL		TRIAL 3
4:30	TRIAL 1	<WEIGHT> <CLEAN&LOAD>	BAKE
5:00	TRIAL 2		
5:30	TRIAL 3	<CLEAN&LOAD> <WEIGHT>	COOL
6:00	BAKE		TRIAL 1
6:30			TRIAL 2
7:00	COOL	<WEIGHT> <CLEAN>	TRIAL 3
7:30	TRIAL 1		
8:00	TRIAL 2		
8:30	TRIAL 3	<CLEAN>	

Figure 6. Optimal schedule for density measurements.

6. DENSITY DATA

Following the procedure laid out in the previous section, the following data was collected for five grass pollens.

COMMON NAME	DRYING				WEIGHT (grams)			DENSITY (g/cm^3)			
	TEMP (°F)	TIME IN	TIME OUT	TOTAL TIME	TOTAL	•	SAMPLE	TRIAL #1	TRIAL #2	TRIAL #3	TRIAL #4
Bahia	225			2:00	20.33738		4.401	1.406	1.406	1.407	--
Bermuda	220	1507	1610	1:03	20.77686	•	4.843	1.434	1.431	1.429	1.426
Blue, Kentucky/June	200	845	1030	1:45	20.55601	•	4.622	1.398	1.397	1.395	1.393
Corn	200	1628	1833	1:07	21.37284		5.437	1.389	1.386	1.384	1.382
Fescue, Meadow	200	1323	1428	2:05	21.68440		5.748	1.419	1.418	1.417	1.417

7. CONCLUSION

The average values and the standard deviation (σ) for the samples are given below. Note that the accuracy of the instrument is ± 0.02 cc. This translates into a standard deviation ($\sigma_{accuracy}$) of 0.010 to 0.013 g/cc which is an order of magnitude greater than the standard deviation ($\sigma_{average}$) calculated from the data below. Therefore the drying technique is acceptable and the measurements accurate to within machine tolerances.

	DENSITY (g/cm^3)						
COMMON NAME	TRIAL #1	TRIAL #2	TRIAL #3	TRIAL #4	AVE	$\sigma_{average}$	$\sigma_{accuracy}$
Bahia	1.406	1.406	1.407	--	1.406	0.001	0.013
Bermuda	1.434	1.431	1.429	1.426	1.430	0.003	0.012
Blue, Kentucky/June	1.398	1.397	1.395	1.393	1.396	0.002	0.012
Corn	1.389	1.386	1.384	1.382	1.385	0.003	0.010
Fescue, Meadow	1.419	1.418	1.417	1.417	1.418	0.001	0.010